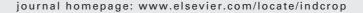
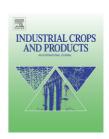


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Chemical constituents and physical properties of guayule wood and bark

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ABSTRACT

About 15 Parthenium species grow in the North American continent with Parthenium argentatum (guayule) as the only species containing harvestable amounts of the rubber latex. The predicted commercialization of the guayule plant for its hypoallergenic latex will result in a significant amount of waste fiber or bagasse biomass that can also be put to use for making wood, paper, and other chemical products, as well as in energy production. Thus, the guayule wood and bark fibers can be considered a new source of plant biomass that may be used as a direct substitute for forest-based wood fiber. However, little information is available on the chemical composition of the wood and bark tissues of guayule (*P. argentatum*). The objectives of this study were to determine the chemical and fiber composition of guayule and to compare it with other wood fiber sources.

Three germplasm lines of mature guayule (Cal-6, AZ-101, and G7-15) and another species of *Parthenium* (*P. tomentosum*, PT), juvenile soft maple (*Acer rubrum*), a deciduous tree, and milkweed (*Asclepias syriaca* L.) that has long fibers were the plant sources. Separate wood and bark tissues were analyzed for hot water, 1% sodium hydroxide, and alcohol–toluene extracts. In addition, the lignin, holocellulose, alpha-cellulose, and pentosan contents were determined.

All the chemical components in the wood fibers for the *Partheniums* were equal to or greater than the juvenile maple tree. Milkweed had higher alpha-cellulose and lower alcohol–toluene extract contents than both the guayule and soft maple. The guayule bark fibers had more chemical extracts than the wood fibers. The specific gravity of guayule wood was greater than the deciduous wood species. However, the fiber lengths of soft maple wood, guayule wood, and milkweed are similar. Based on the chemical composition, *P. argentatum* and *P. tomentosum* could serve as raw materials for the paper and chemical industries as well as for energy production.

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1. Introduction

Approximately 15 species of Parthenium are native to the North American continent, and of these, P. argentatum (guayule) is

the only species with harvestable amounts of rubber latex (Hammond and Polhamus, 1965). Milkweed (Asclepias syriaca), a noxious weed growing in cultivated crops, produces a small amount of latex, but is mainly used for the production of down

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fiber for insulation (Knudsen and Zeller, 1991). The maple tree is a deciduous species that grows on a wide range of sites and is used in many types of wood products (Bowyer and Haygreen, 2003).

As the predicted commercialization of the guayule plant for its hypoallergenic latex progresses, a large amount of waste fiber or bagasse will be produced and use must be made of this biomass to make latex production economically profitable. This bagasse fiber can be used for making termite resistant wood products (Nakayama et al., 2001). Other potential applications include fabrication of paper, chemical derivatives, and energy production (Touzinsky, 1980; Nakayama et al., 2003; Holt et al., 2005). Thus, the guayule wood and bark fibers can be considered as new and alternative sources of plant biomass that may be used as a renewable resource for direct substitution of the diminishing forest-based wood sources. However, little information is available on the chemical composition of the wood and bark tissues of guayule.

The principal constituents of wood are the polysaccharides, lignin, and extracts. Guayule biomass possibly can replace or supplement traditional sources of raw materials such as petroleum and timber. Although guayule is associated as a source of rubber, the plant biomass may emerge as an important source of paper and pulp, fiber composition, and chemicals for the future (Nakayama, 2005).

A wide range of organic chemicals and fuel can be produced from the basic lignocellulose structure of woody biomass products that are obtainable from pentose, cellulose, and lignin. The pentose can be converted into useful products by fermentation (vitamin, protein, and fat), dehydration (furfural), hydrogenation (polyols), and crystallization (xylose). In a similar manner, cellulose can be converted into alcohols, glycerols, ketones, and acids by fermentation; levulinic acid and hydroxymethyl furfural by hydrolysis and dehydration; and phenolics (hydrolysis) and aromatics (hydrogenation) from lignin. Synthetic fibers and polymers such as nylon and rayon can also be derived from the alpha-cellulose, and vanillin and adhesives from lignin (Goldstein, 1981).

The extracts of wood and bark in living plants mainly serve as food reserves (fat, fatty acids, sugars, and starch), protectants (terpenes, resin acids, and phenols) and plant hormones (phytosterols). These can be extracted using suitable solvents. The recommended three ASTM standard extraction

test methods for woody biomass are based on hot water, aqueous sodium hydroxide (1%), and an alcohol-toluene mixture. The principal compounds extracted with these solvents are:

- 1. Hot water: inorganic salts, oligosaccharides, sugars, cycloses, cyclitols, and phenolics.
- 2. NaOH (1%): fatty acids, wax, resin, essential oils, lignin, and hemicelluloses.
- Alcohol-toluene: certain phenolic substances (lignin fraction, sterols, tannins, and phlobaphenes), some organic acids (resin acid, amino acids, vanillic acid, and syringic acid), and other materials (such as pigments and syringaldehyde).

The physical properties and chemical components of wood profoundly influence its utilization. Guayule plant biomass may have potential for replacing or supplementing other raw materials for making commercial products. Because guayule is drought tolerant and has low fertility requirement, it has potential for commercial production for the semiarid regions.

The primary objective of this study was to determine the chemical composition and physical nature of the wood and bark of three guayule lines and a related species. In addition, the stems of milkweed and maple were included for comparative purposes.

2. Materials and methods

2.1. Materials and sample preparation

Three germplasm lines of 3-year-old guayule plants (Cal-6, AZ-101, and G7-15) and P. tomentosum were harvested in 2002. All leaves were removed and only branches greater than about 0.6 cm were separated. Milkweed was obtained from Natural Fiber Corp., Ogallala, NE. The 2-year-old red maple trees were sampled from the bottomland site located in southern Illinois at the University of Illinois, Dixon Springs Agricultural Center. A breakdown of the samples used is listed in Table 1.

The guayule bark tissue was separated manually from the stem and then air-dried and milled. The coarse material was ground in a Wiley mill to pass a no. 40 (425- μ m) mesh screen and then a no. 60 (250- μ m) mesh screen for the vari-

Sample	Replicates	Number of plants	Total air-dry weight (g) & moisture (%)	Total milled dry weight (g)		
				Wood	Bark	
Cal-6	А	3	881 (56.67%) ^a	146	213	
	В	3	901 (49.25%)	173	188	
AZ-101	Α	2	1141 (56.38%)	197	274	
	В	2	1079 (57.80%)	239	180	
G7-15	Α	1	1326 (43.86%)	304	271	
	В	1	1625 (43.12%)	368	281	
P. tomentosum	Α	1	3094 (80.0%)	1,101	267	
	В	1	2864 (78.0%)	907	256	
Guayule bagasse	_	_	_ ′	1,050		
Milkweed stems	_	_	673 (10.2%)	108	283	

^a Values in parenthesis are the moisture content in percent based on oven-dry weight.

Plant sources	Reps	Stem moisture content (%)	Stem specific gravity	Stem fiber length (mm)	Extract contents						
					NaOH (1%) (%)		Alcohol–toluene (%) wood bark		Hot water (%) wood bark		
					Wood	Bark	Wood	Bark	Wood	Bark	
Cal-6	Α	56.68	0.691	0.394	33.10	56.02	6.57	22.03	10.34	15.39	
		(10.2) ^b	(13.5)	(7.7)	(0.36)	(1.37)	(3.81)	(2.56)	(3.97)	(2.19)	
	В	49.25	0.712	0.387	29.16	54.48	4.52	18.29	7.30	16.71	
		(12.7)	(3.30)	(6.41)	(0.64)	(0.72)	(8.79)	(0.79)	(3.28)	(5.82)	
AZ101	Α	56.38	0.672	0.466	33.47	56.15	7.86	26.85	12.81	22.25	
		(0.80)	(3.50)	(8.48)	(0.39)	(4.38)	(1.91)	(2.29)	(11.9)	(3.20)	
	В	57.80	0.693	0.394	29.71	57.18	6.35	26.81	9.73	21.90	
		(19.7)	(4.25)	(2.79)	(0.20)	(3.59)	(3.56)	(1.67)	(2.56)	(10.41)	
G7-15	Α	43.86	0.725	0.481	28.29	58.96	5.46	22.44	7.97	18.25	
		(15.8)	(3.40)	(16.9)	(0.94)	(9.26)	(5.45)	(3.97)	(1.72)	(20.7)	
	В	43.12	0.801	0.452	30.13	57.55	4.48	26.41	9.76	22.49	
		(32.0)	(3.80)	(6.19)	(10.7)	(0.36)	(11.8)	(0.93)	(1.63)	(0.96)	
P. tomentosum	Α	59.90	0.725	0.469	19.17	70.62	2.78	18.11	6.40	33.95	
		(19.0)	(6.00)	(13.8)	(0.97)	(3.23)	(16.3)	(5.98)	(4.99)	(2.07)	
	В	74.27	0.666	0.411	19.48	68.61	3.60	16.93	6.61	37.71	
		(55.9)	(13.3)	(11.5)	(2.22)	(1.69)	(12.7)	(6.05)	(3.04)	(0.51)	
Bagasse (P. argentatum)		12.50	-	-	51.83		14.98		25.88		
		(2.0)			(1.09)		(1.29)		(13.64)		
Milkweed		10.2	0.374	0.406	31.30	47.87	2.752	7.71	10.31	24.80	
		(0.5)	(20.0)	(15.8)	(3.66)	(2.14)	(10.5)	(22.0)	(1.63)	(3.58)	
Red maple (2-year)		75.50	0.400	0.520	17.10	34.15	4.63	12.84	7.46	28.15	
,		(10.5)	(10.0)	(5.6)	(1.59)	(1.24)	(3.82)	(1.99)	(3.52)	(1.22)	

^a Each value is an average of at least three tests except that each fiber length value is an average for 65 measurements of fiber samples using TAPPI standard grid-count technique (TAPPI, 1980).

^b The value in parenthesis is the coefficient of variation CV (%) = (S.D./x)(100).

ous analyses. The bark materials had to be pre-frozen before the grinding process in order to avoid the gumming of the screen by the resinous material present in guayule bark. The guayule bagasse was the waste residual obtained from the water–solvent extraction of latex from the whole plant.

2.2. Test methods and analysis

The stem sections were analyzed for specific gravity, fiber length, and moisture content. For the fiber length determination, a projection method was used following the Technical Association of Pulp and Paper Industry (TAPPI) standard and the suggested grit-count method (TAPPI, 1980). All analyses and procedures followed the methods described in the standards of the American Society for Testing and Materials (Chow and Lucas, 1988; ASTM, 1993; Chow et al., 1995). ASTM test methods (1993) were also used to determine the ash (D1102), alpha-cellulose (D1103), holocellulose (D1104, modified), lignin (D1106), alcohol-toluene (D1107), NaOH (D1109), and hot-water (D11110) extract contents of all samples. Pentosan content was determined with the T-19m test method described in the standards of the Technical Association of Pulp and Paper Industry (TAPPI, 1992). Analysis of variance was conducted to determine the effects of the three-germplasm lines of guayule and P. tomantosum tissue portion (wood vs. bark) on the chemical properties.

2.3. Experimental design

The randomized-complete-block design was used for testing the chemical and physical properties. Duncan's multiple-range test and t-test for detecting the difference in properties between line and tissue portion were also used (Steel and Torrie, 1980; SAS, 1989). These included the four *Parthenium*, the milkweed, and maple plant parts consisting of the wood and bark. Interactions were examined for each independent variable. A linear model was used for explaining sources of variation.

3. Results and discussion

The specific gravities of woody stems ranged from 0.666 to 0.801 g/cc for the three guayule lines and P. tomentosum, 0.40 for the maple, and 0.37 for the milkweed sample (Table 2). The guayule specific gravity is higher than most wood species. The average wood specific gravity of tree species ranges from 0.30 to 0.75 in the United States. The specific gravity serves as a valuable indicator of the amount of wood pulp, chemicals, and energy that could be obtained from the wood.

The fiber lengths of these stems ranged from 0.387 to 0.481 mm for the three guayule lines and P. tomentosum, 0.520 mm for the maple that is the representative of the common deciduous tree species in the United States, and

Plant sources	Reps	Ash content (%)		Holocellulose content (%)		Alpha-cellulose content (%)		Lignin content (%)		Pentosan content (%)	
		Wood	Bark	Wood	Bark	Wood	Bark	Wood	Bark	Wood	Bark
Cal-6	А	2.495	6.222	78.79	67.81	44.78	39.83	21.64	32.80	23.63	11.30
		$(0.17)^{b}$	(0.42)	(0.70)	(0.10)	(2.14)	(10.5)	(5.38)	(0.84)	(2.79)	(3.49)
	В	2.108	5.752	79.93	65.55	46.88	42.51	19.95	34.48	23.94	13.24
		(0.21)	(0.15)	(0.30)	(1.10)	(0.53)	(5.64)	(3.96)	(1.45)	(1.41)	(2.79)
AZ-101	Α	1.900	6.461	79.41	70.49	46.75	47.78	19.82	36.98	19.86	9.66
		(0.29)	(0.58)	(0.30)	(1.00)	(1.24)	(8.48)	(8.25)	(1.26)	(2.65)	(3.47)
	В	1.903	6.377	79.95	64.86	48.94	37.07	20.60	35.25	19.96	10.60
		(0.48)	(1.06)	(0.10)	(0.20)	(1.02)	(4.99)	(5.20)	(3.95)	(1.79)	(7.99)
G7-15	Α	1.978	5.968	81.10	60.44	45.27	35.22	22.68	42.60	22.74	9.88
		(0.81)	(0.49)	(0.90)	(0.30)	(1.06)	(4.56)	(1.72)	(0.54)	(1.29)	(6.66)
	В	1.453	5.972	82.21	60.75	46.71	32.52	20.33	34.59	21.62	10.84
		(0.95)	(0.50)	(0.50)	(1.60)	(1.62)	(7.44)	(4.43)	(88.0)	(2.14)	(5.26)
P. tomentosum	Α	1.713	7.664	82.24	52.54	50.15	30.66	17.41	24.90	15.83	13.37
		(0.97)	(0.74)	(0.30)	(1.10)	(0.50)	(5.91)	(5.41)	(4.75)	(1.08)	(2.56)
	В	1.645	7.041	83.56	49.46	51.03	29.15	19.88	23.27	16.00	12.54
		(0.48)	(0.47)	(0.20)	(0.10)	(1.80)	(5.38)	(3.40)	(6.93)	(2.01)	(2.90)
Bagasse (P. argentatum)		9.839		62.60		41.38		25.29		10.95	
		(0.19)		(0.60)		(1.36)		(1.25)		(4.61)	
Milkweed		4.554	12.716	76.49	69.75	72.50	43.54	21.79	8.48	21.54	8.81
		(0.45)	(0.65)	(0.20)	(0.80)	(0.59)	(0.17)	(1.78)	(7.56)	(2.24)	(0.96)
Red maple		0.990	4.810	67.35	62.54	37.71	31.87	26.03	33.10	22.05	18.98
(2-year)		(1.18)	(0.93)	(0.30)	(0.80)	(0.70)	(1.10)	(2.32)	(1.07)	(1.75)	(3.52

^a Each value is the mean of three tests, except alpha-cellulose in bark for the four Parthenium types.

b The number in parenthesis is the coefficient of variation, CV(%) = (S.D./x)(100).

 $0.406\,\mathrm{mm}$ for the wood core fiber of milkweed stems (Table 2). The length of the bark or bast fiber of milkweed exceeded 2 mm.

The extract content of the bark fraction is much higher than the wood fraction (Table 2). The high values for the bagasse extract content are caused by the residual sodium sulfite, which is used as an antioxidant in the latex extraction process. In the wood portion of the four *Parthenium* lines, the AZ-101 and *P. tomentosum* had the highest and the lowest three extract contents, respectively. The alcohol-toluene extract content in guayule bark also exhibited the same trend. In the bark portion of the four *Parthenium* types, the *P. tomentosum* and Cal-6 had

the highest and the lowest hot-water extract and NaOH extract contents, respectively. Both the maple and milkweed had relatively low alcohol-toluene extractive contents, but their bark portion had a moderate amount of two other kinds of the extractives.

The bark portion of all samples had higher ash and lignin contents than those of the wood portion except for the milk-weed (Table 3). On the other hand, the wood portion of all samples possessed higher holocellulose, alpha-cellulose, and pentosan contents than the bark. The ash content of the guayule bagasse is high, as noted previously in the extraction data because of the residual salts from the latex extraction

Properties	Stem part		Bagasse	Mlkw	Maple			
		Н	igh	Lo	DW W			
Alcohol–toluene e	xtracts							
	Wood	AZ-101	>Cal-6	=G7-15	>P. tomentosum		2.75	4.63
		(7.11)	(5.55)	(4.97)	(3.19)	14.9		
	Bark	AZ-101	>G7-15	>Cal-6	>P. tomentosum		7.71	12.8
		(26.8)	(24.4)	(20.16)	(17.52)			
Hot-water extracts			07.45	0.1.6	.		7.46	
	Wood	AZ-101	>G7-15	=Cal-6	>P. tomentosum	25.0	7.46	7.5
	Bark	(11.61)	(8.86) >AZ-101	(8.80)	(6.62) >Cal-6	25.9	28.15	28.1
	Bark	P. tomentosum (35.83)	(22.08)	=G7-15 (20.31)	(16.09)		28.15	28.1
		(33.63)	(22.08)	(20.31)	(10.09)			
NaOH extracts								
	Wood	AZ-101	=Cal-6	>G7-15	=P. tomentosum		31.3	17.0
	D 1	(31.59)	(31.13)	(29.21)	(19.33)	51.8	47.0	
	Bark	P. tomentosum	>G7-15	>AZ-101	>Cal-6		47.8	34.1
		(69.61)	(58.36)	(56.66)	(54.78)			
Ash content	Wood	G7-15	=Cal-6	=AZ-101	>P. tomentosum		4.5	0.9
		(2.45)	(2.25)	(1.90)	(1.68)	9.8		
	Bark	P. tomentosum	>AZ-101	=Cal-6	=G7-15		12.7	4.8
		(7.35)	(6.41)	(5.98)	(5.97)			
Lignin content	Wood	G7-15	=Cal-6	=AZ-101	>P. tomentosum		21.8	27.0
· ·		(21.11)	(20.79)	(20.21)	(18.65)	25.3		
	Bark	G7-15	>AZ-101	>Cal-6	>P. tomentosum		8.5	33.0
		(38.59)	(36.12)	(33.54)	(24.39)			
Holocellulose	Wood	P. tomentosum	>G7-15	>Cal-6	=AZ-101		76.5	67.0
Holoccifalobe	wood	(80.33)	(77.79)	(75.20)	(74.06)	62.6	70.5	07.0
	Bark	Cal-6	>AZ-101	>G7-15	>P. tomentosum		69.7	62.0
		(55.49)	(53.35)	(48.71)	(43.39)			
Alpha-cellulose	Wood	P. tomentosum	>AZ-101	>G7-15	=Cal-6		72.5	37.7
Alpha-cellulose	wood	(50.59)	(47.84)	(46.00)	(45.83)	41.3	72.5	3/./
	Bark	(30.39) Cal-6	>AZ-101	>G7-15	>P. tomentosum	41.5	43.5	31.0
	Dark	(41.17)	(38.93)	(36.87)	(29.90)		15.5	31.0
		, ,	, , ,	, ,	, ,			
Pentosan	Wood	Cal-6	>G7-15	>AZ-101	>P. tomentosum	400	21.5	22.0
	D 1	(23.78)	(22.18)	(19.90)	(15.91)	10.9	0.0	40.0
	Bark	P. tomentosum	=Cal-6	>G7-15	=AZ-101		8.8	18.0
		(12.95)	(12.50)	(10.36)	(10.12)			
Specific gravity	Stem	G7-15	>Cal-6	=P. tomentosum	>AZ-101	-	0.37	0.40
		(0.76)	(0.70)	(0.70)	(0.68)			
Fiber length (mm)	Stem	G7-15	>P. tomentosum	=AZ-101	>Cal-6	_	0.40	0.52
	0.0111	(0.47)	(0.44)	(0.43)	(0.39)		0.10	3.32

Significance level, 0.05; left to right are from highest to lowest; bagasse, guayule bagasse; mlkw, milkweed stem and maple, 2-year-old maple tree; the value in parenthesis is the coefficient of variation, CV (%) = (S.D./x)(100).

processing. Because the bagasse is the ground mixture of wood and bark, its wood composition was intermediate compared with the other *Parthenium* samples.

The chemical and physical properties for wood and bark fiber tissues are compared in Table 4 for all the plant sources analyzed. In the preliminary statistical analysis, the guayule replicate (A vs. B) was found not to be a highly significant factor affecting all tested properties. Thus, these values for all the properties were combined. In the analysis of variance, both factors of plant portions (wood vs. bark) and four *Parthenium* types were different in chemical properties. The bark for the four types had higher ash, all of the three kinds of extracts, and lignin contents than its equivalent wood portion. On the other hand, the *Parthenium* wood samples had higher pentosan, holocellulose, and alpha-cellulose contents than the bark material.

The bagasse, a mixture of wood and bark, had chemical compositions intermediate to the wood portion of the four *Parthenium* types. The 2-year-old maple wood portion had the highest lignin, the lowest ash, a relatively low extract (alcohol-toluene, hot water, and NaOH), holocellulose, and alpha-cellulose contents. The milkweed stems had the lowest alcohol-toluene extracts, the highest alpha-cellulose content in the wood portion, and the highest holocellulose content in the bark tissue that were comparable with those obtained for the four *Parthenium* types. In addition, the guayule AZ-101 wood, and bark of *P. tomentosum*, G7-15, and Cal-6 had high extract contents based on the three extraction methods.

The results from this study show that these four types of *Parthenium* sources and their bagasse have physical and chemical composition which have potential for use in the pulp and paper and chemical industries as well as for fuel and energy sources. Thus, they can be used for direct substitution of forest-based biomass materials.

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